

IN THE SPECIFICATION

Please amend page 1 by inserting the following heading between the title of the invention and the first paragraph:

“FIELD OF THE INVENTION”

Please amend page 1 by inserting the following heading between the first and second paragraphs:

“BACKGROUND OF THE INVENTION”

Please amend page 3 by inserting the following heading on line 3:

“BRIEF SUMMARY OF THE INVENTION”

Please amend page 7 by inserting the following heading on line 28:

“BRIEF DESCRIPTION OF THE DRAWINGS

Preferred features of the present invention...”

Please amend page 8 by inserting the following heading on line 11:

“DETAILED DESCRIPTION OF THE INVENTION

In the present invention, one can achieve...”

Please amend page 17 by inserting the following language between the “CLAIMS” heading and claim 1:

“I/We claim:”

Please amend pages 3-5 by canceling the following text beginning on page 3 line 9 and ending on page 5 line 19:

~~“The present invention thus provides a simple and convenient technique for forming an anti-corrosive coating on a plastics component of a vacuum pump. By the term “anti-corrosive” it should be understood to mean that the coating is capable of withstanding wear and degradation as a result of exposure to abrasive particles and gases such as fluorine, chlorine-trifluoride, tungsten-hexafluoride, chlorine, boron-trichloride, hydrogen bromide, oxygen and~~

the like. The coating can be conveniently formed from any suitable barrier layer forming metal or alloy thereof.

By the term "barrier layer forming metal" it should be understood to mean those metals and their alloys (such as Al, Mg, Ti, Ta, Zr, Nb, Hf, Sb, W, Mo, V, Bi), the surfaces of which naturally react with elements of the environment in which they are placed (such as oxygen) to form a coating layer, which further inhibits the reaction of the metal surface with said reactive environmental elements.

The technique of electrolytic plasma oxidation (EPO) is known by various other names, for example anodic plasma oxidation (APO), anodic spark oxidation (ASO), micro-arc oxidation (MAO). During this technique, a partial oxygen plasma forms at the metal/gas/electrolyte phase boundary and results in the creation of a ceramic oxide layer. The metal ion in the ceramic oxide layer is derived from the metal and the oxygen formed during the anodic reaction of the aqueous electrolyte at the metal surface. At temperatures of 7000K associated with the formation of the plasma, the ceramic oxide exists in a molten state. This means that the molten ceramic oxide can achieve intimate contact with the metal surface at the metal/oxide boundary, which means that the molten ceramic oxide has sufficient time to contract and form a sintered ceramic oxide layer with few pores. At the electrolyte/oxide boundary, however, the molten ceramic oxide is quickly cooled by the electrolyte and the gases flowing away, notably oxygen and water vapour, leaving an oxide ceramic layer with increased porosity.

Thus, the ceramic oxide coating so formed is itself characterised by three layers or regions. The first is a transitional layer between the metallic layer and the coating where the metal surface has been transformed, resulting in excellent adhesion for the coating. The second is the functional layer, comprising a sintered ceramic oxide containing hard crystallites that give the coating its high hardness and wear resistance characteristics. The third is the surface layer, which has lower hardness and higher porosity than the functional layer.

It will be appreciated from the foregoing that the ceramic oxide coating is atomically bound to the underlying metallic layer and is formed from the surface of the metallic layer. This means that the ceramic oxide coating so produced exhibits greater adhesion to the underlying metallic layer than would be formed from externally applied sprayed ceramic coating. The

~~ceramic oxide coating exhibits superior surface properties such as extreme hardness, very low wear, detonation and cavitation resistance, good corrosion and heat resistance, high dielectric strength and a low coefficient of friction. In addition, it is also resistant to corrosion from halogens, inter-halogen compounds and other semiconductor processing chemicals excited by plasma.~~

~~From the foregoing it will be appreciated that the external surface of the coating is in some applications characterised by a low porosity. In such situations out-gassing from the coated substrate material is minimised. In other applications, the external surface of the coating may be irregular and exhibit some porosity. In order to ensure extreme hardness, low wear and good corrosion resistance, the external surface of this coating may be removed by grinding to expose the underlying sintered ceramic oxide layer, which provides the superior surface properties referred to above.~~

~~Alternatively, where the external surface of the coating exhibits some porosity it can serve as a matrix for application of an optional layer of a composite nature. In such situations, materials suitable for forming the composite layer include a lubricant or paint, for example. It will be appreciated that the pore sizes of the external surface of the second layer are of a size that are capable of retaining the material of the third layer. Other examples of such composite coatings include lubricants such as fluorocarbons, polytetrafluoroethylene (PTFE), molybdenum disulfide (MoS₂), graphite and the like, which are retained by the porous external surface of the coating. The optional layer is preferably formed directly over the coating, the coating providing a key for the adhesion of this additional layer.~~

~~In one embodiment, the metallic layer is not formed directly on the surface of the substrate, but is formed on the surface of a metallic layer previously applied to the substrate. Applying this metallic layer, formed, for example, from nickel, on the surface of the substrate can improve the properties of the surface on which the subsequent metallic layer is deposited. Furthermore, a coating formed from nickel, aluminium, and ceramic oxide layers would offer superior corrosion, wear resistance and heat transfer capability to a metallic substrate, such as an aluminium alloy used in the manufacture of high-speed vacuum pumps.'~~

Please amend pages 5-7 by canceling the following text beginning on page 5 line 26

and ending on page 7 line 27:

~~“The (second) metallic layer is suitably applied by depositing a layer of the barrier layer-forming metal or alloy thereof directly or indirectly (depending on substrate) onto the substrate surface to a thickness of preferably less than 100um. The metallic layer is preferably deposited onto the surface of the substrate using one of (i), sifting or compression of metallic powder or wrapping of the foil onto a liquid adhesive, after it has been applied to the surface (ii), electrolytic deposition onto an initially deposited metal layer (iii), spraying techniques such as sputtering, plasma spraying, arc spraying, flame spraying, vacuum metallising, ion-vapour deposition, high velocity oxyfuel spraying, cold gas spray; combinations thereof and the like, which are well known to a skilled person. These methods ensure that the metal or alloy thereof is both well adhered to and does not degrade the underlying substrate. Whatever procedure or combination thereof adopted, the parameters must be adjusted to values suitable to obtain homogeneous coatings, with low porosity value and free of cast-in (embedded) particles, oxides and cracks that will compromise the formation of the ceramic oxide coating by electrolytic plasma oxidation. For both metal and plastic substrates, the deposition of a metallic layer on the surface of the substrate has little effect on the bulk temperature of the substrate, thereby preventing distortion thereof. When employing the hot spraying techniques, the superior wetting properties of the molten metal particles on the substrate surface, when compared to conventionally sprayed ceramic particles, lead to the formation of a metallic layer having a low porosity.~~

~~As indicated above, the coating is formed by electrolytic plasma oxidation of the surface of the metallic layer. The coating is suitably formed by immersing an anodically charged metal coated part in an alkaline electrolyte (e. g., aqueous solution of an alkali metal hydroxide and sodium silicate) using a stainless steel bath acting as the counter electrode and applying an AC voltage in excess of 250V to the part. During this technique, a partial oxygen plasma forms at the metal/gas/electrolyte phase boundary and results in the creation of a ceramic oxide layer. The metal ion in the ceramic oxide layer is derived from the metal and the oxygen formed during the anodic reaction of the aqueous electrolyte at the metal surface. At temperatures of 7000K associated with the formation of the plasma, the ceramic oxide exists in a molten state. This means that the molten ceramic oxide can achieve intimate contact with the metal surface at the metal/oxide boundary, which means that the molten ceramic oxide has sufficient time to~~

~~contract and form a sintered ceramic oxide layer with few pores. At the electrolyte/oxide boundary, however, the molten ceramic oxide is quickly cooled by the electrolyte and the gases flowing away, notably oxygen and water vapour, leaving an oxide ceramic layer with increased porosity. The bath temperature is maintained constant at about 20°C. A constant current density of at least 1A/dm is maintained in the electrolytic bath until the voltage reaches a predetermined end value, consistent with the formation of an insulating layer. Under these conditions, one obtains typically about 1 µm of ceramic oxide coating per minute. Ceramic coating thickness up to about 100µm can be obtained in 60 minutes, depending on barrier forming metal type and alloy. The required current density to initiate the plasma process may be as high as 25A/dm² if the applied metallic layer is rough and porous.~~

~~The electrolytic plasma oxidation is preferably carried out in a weak aqueous alkaline electrolyte of pH in the range from 7 to 8.5, preferably in the range from 7.5 to 8, at temperatures of about 20°C, which means that the integrity of the substrate material is little affected. As indicated above the melting that occurs during the formation of the ceramic coating tends to fill out any pores in the underlying metallic layer, resulting in an impermeable interfacial region between the layers.~~

~~For plastic substrates the formation of the ceramic oxide coating over the underlying metallic layer overcomes the problems of electrostatic repulsion commonly encountered when depositing ceramic particles directly onto the surfaces of plastic substrates.~~

~~The substrate is preferably a component of a vacuum pump, and so the present invention also provides a vacuum pump component formed from metallic or plastics material and having a coating thereon formed by electrolytic plasma oxidation of a metallic layer applied to the component."~~

Please amend page 10 by inserting the following text on line 16:

"The present invention thus provides a simple and convenient technique for forming an anti-corrosive coating on a plastics component of a vacuum pump. By the term "anti-corrosive" it should be understood to mean that the coating is capable of withstanding wear and degradation as a result of exposure to abrasive particles and gases such as fluorine, chlorine-

trifluoride, tungsten-hexafluoride, chlorine, boron-trichloride, hydrogen bromide, oxygen and the like. The coating can be conveniently formed from any suitable barrier layer-forming metal or alloy thereof.

By the term "barrier layer-forming metal" it should be understood to mean those metals and their alloys (such as Al, Mg, Ti, Ta, Zr, Nb, Hf, Sb, W, Mo, V, Bi), the surfaces of which naturally react with elements of the environment in which they are placed (such as oxygen) to form a coating layer, which further inhibits the reaction of the metal surface with said reactive environmental elements.

The technique of electrolytic plasma oxidation (EPO) is known by various other names, for example anodic-plasma oxidation (APO), anodic spark oxidation (ASO), micro-arc oxidation (MAO). During this technique, a partial oxygen plasma forms at the metal/gas/electrolyte phase boundary and results in the creation of a ceramic oxide layer. The metal ion in the ceramic oxide layer is derived from the metal and the oxygen formed during the anodic reaction of the aqueous electrolyte at the metal surface. At temperatures of 7000K associated with the formation of the plasma, the ceramic oxide exists in a molten state. This means that the molten ceramic oxide can achieve intimate contact with the metal surface at the metal/oxide boundary, which means that the molten ceramic oxide has sufficient time to contract and form a sintered ceramic oxide layer with few pores. At the electrolyte/oxide boundary, however, the molten ceramic oxide is quickly cooled by the electrolyte and the gases flowing away, notably oxygen and water vapour, leaving an oxide ceramic layer with increased porosity.

Thus, the ceramic oxide coating so formed is itself characterised by three layers or regions. The first is a transitional layer between the metallic layer and the coating where the metal surface has been transformed, resulting in excellent adhesion for the coating. The second is the functional layer, comprising a sintered ceramic oxide containing hard crystallites that give the coating its high hardness and wear resistance characteristics. The third is the surface layer, which has lower hardness and higher porosity than the functional layer.

It will be appreciated from the foregoing that the ceramic oxide coating is atomically bound to the underlying metallic layer and is formed from the surface of the metallic layer. This means

that the ceramic oxide coating so produced exhibits greater adhesion to the underlying metallic layer than would be formed from externally applied sprayed ceramic coating. The ceramic oxide coating exhibits superior surface properties such as extreme hardness, very low wear, detonation and cavitation resistance, good corrosion and heat resistance, high dielectric strength and a low coefficient of friction. In addition, it is also resistant to corrosion from halogens, inter-halogen compounds and other semiconductor processing chemicals excited by plasma.

From the foregoing it will be appreciated that the external surface of the coating is in some applications characterised by a low porosity. In such situations out-gassing from the coated substrate material is minimised. In other applications, the external surface of the coating may be irregular and exhibit some porosity. In order to ensure extreme hardness, low wear and good corrosion resistance, the external surface of this coating may be removed by grinding to expose the underlying sintered ceramic oxide layer, which provides the superior surface properties referred to above.

Alternatively, where the external surface of the coating exhibits some porosity it can serve as a matrix for application of an optional layer of a composite nature. In such situations, materials suitable for forming the composite layer include a lubricant or paint, for example. It will be appreciated that the pore sizes of the external surface of the second layer are of a size that are capable of retaining the material of the third layer. Other examples of such composite coatings include lubricants such as fluorocarbons, polytetrafluoroethylene (PTFE), molybdenum disulfide (MoS₂), graphite and the like, which are retained by the porous external surface of the coating. The optional layer is preferably formed directly over the coating, the coating providing a key for the adhesion of this additional layer.

In one embodiment, the metallic layer is not formed directly on the surface of the substrate, but is formed on the surface of a metallic layer previously applied to the substrate. Applying this metallic layer, formed, for example, from nickel, on the surface of the substrate can improve the properties of the surface on which the subsequent metallic layer is deposited. Furthermore, a coating formed from nickel, aluminium, and ceramic oxide layers would offer superior corrosion, wear resistance and heat transfer capability to a metallic substrate, such as an aluminium alloy used in the manufacture of high speed vacuum pumps.

The (second) metallic layer is suitably applied by depositing a layer of the barrier layer-forming metal or alloy thereof directly or indirectly (depending on substrate) onto the substrate surface to a thickness of preferably less than 100um. The metallic layer is preferably deposited onto the surface of the substrate using one of (i), sifting or compression of metallic powder or wrapping of the foil onto a liquid adhesive, after it has been applied to the surface (ii), electrolytic-deposition onto an initially deposited metal layer (iii), spraying techniques such as sputtering, plasma-spraying, arc-spraying, flame-spraying, vacuum-metallising, ion-vapour deposition, high velocity oxyfuel-spraying, cold gas-spray; combinations thereof and the like, which are well known to a skilled person. These methods ensure that the metal or alloy thereof is both well adhered to and does not degrade the underlying substrate. Whatever procedure or combination thereof adopted, the parameters must be adjusted to values suitable to obtain homogeneous coatings, with low porosity value and free of cast-in (embedded) particles, oxides and cracks that will compromise the formation of the ceramic oxide coating by electrolytic plasma oxidation. For both metal and plastic substrates, the deposition of a metallic layer on the surface of the substrate has little effect on the bulk temperature of the substrate, thereby preventing distortion thereof. When employing the hot spraying techniques, the superior wetting properties of the molten metal particles on the substrate surface, when compared to conventionally sprayed ceramic particles, lead to the formation of a metallic layer having a low porosity.

As indicated above, the coating is formed by electrolytic plasma oxidation of the surface of the metallic layer. The coating is suitably formed by immersing an anodically charged metal coated part in an alkaline electrolyte (e. g. , aqueous solution of an alkali metal hydroxide and sodium silicate) using a stainless steel bath acting as the counter electrode and applying an AC voltage in excess of 250V to the part. During this technique, a partial oxygen plasma forms at the metal/gas/electrolyte phase boundary and results in the creation of a ceramic oxide layer. The metal ion in the ceramic oxide layer is derived from the metal and the oxygen formed during the anodic reaction of the aqueous electrolyte at the metal surface. At temperatures of 7000K associated with the formation of the plasma, the ceramic oxide exists in a molten state. This means that the molten ceramic oxide can achieve intimate contact with the metal surface at the metal/oxide boundary, which means that the molten ceramic oxide has sufficient time to contract and form a sintered ceramic oxide layer with few pores. At the electrolyte/oxide

boundary, however, the molten ceramic oxide is quickly cooled by the electrolyte and the gases flowing away, notably oxygen and water vapour, leaving an oxide ceramic layer with increased porosity. The bath temperature is maintained constant at about 20°C. A constant current density of at least 1 A/dm is maintained in the electrolytic bath until the voltage reaches a predetermined end value, consistent with the formation of an insulating layer. Under these conditions, one obtains typically about 1 µm of ceramic oxide coating per minute. Ceramic coating thickness up to about 100µm can be obtained in 60 minutes, depending on barrier forming metal type and alloy. The required current density to initiate the plasma process may be as high as 25 A/dm² if the applied metallic layer is rough and porous.

The electrolytic plasma oxidation is preferably carried out in a weak aqueous alkaline electrolyte of pH in the range from 7 to 8.5, preferably in the range from 7.5 to 8, at temperatures of about 20°C, which means that the integrity of the substrate material is little affected. As indicated above the melting that occurs during the formation of the ceramic coating tends to fill out any pores in the underlying metallic layer, resulting in an impermeable interfacial region between the layers.

For plastic substrates the formation of the ceramic oxide coating over the underlying metallic layer overcomes the problems of electrostatic repulsion commonly encountered when depositing ceramic particles directly onto the surfaces of plastic substrates.

The substrate is preferably a component of a vacuum pump, and so the present invention also provides a vacuum pump component formed from metallic or plastics material and having a coating thereon formed by electrolytic plasma oxidation of a metallic layer applied to the component.”